

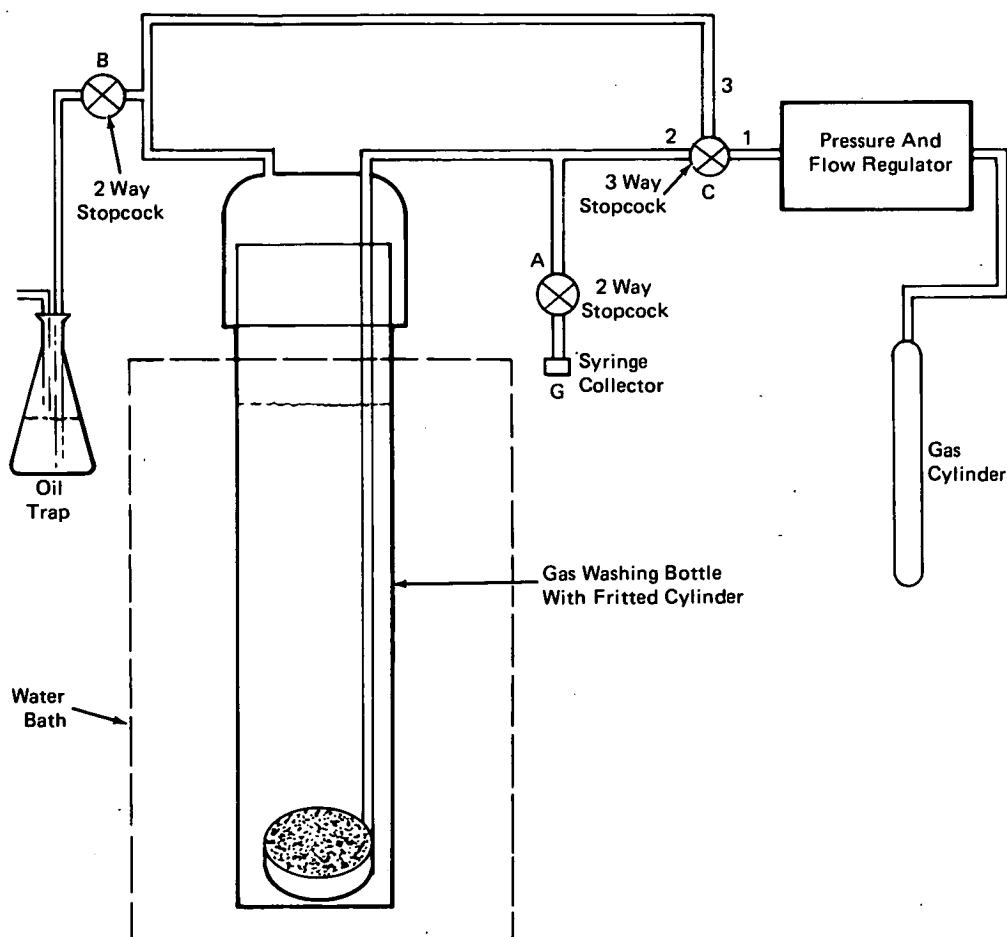
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Marshall Space Flight Center



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Calibration of Dissolved Oxygen Standard for Analysis with Methylene Blue



Apparatus for Preparing Dissolved Oxygen Standard

In the presence of an amino carboxylic acid, a solution of methylene blue (an organic dye) is quantitatively reduced by dissolved oxygen to the colorless leuco form. This reaction has not been widely used for oxygen analysis because of the lack of an accurate method of calibration. Furthermore, this problem exists with other techniques for measuring dissolved oxygen.

The preparation of a solution with a known quantity of dissolved O_2 is complicated by several factors; atmospheric exposure may lead to the absorption and desorption of dissolved gases, and the response characteristics of the measurement system may vary with salinity, temperature, and concentration.

(continued overleaf)

Accurate standard solutions of oxygen can be prepared with the apparatus shown in the figure. Water of the desired salinity is added to the washing bottle, and the water bath is adjusted to the particular temperature of interest. The standard gas mixture is flowed through the system at 50 ml/min by adjusting valve C for flow in the direction 1 to 2, closing A, and opening B. When equilibrium is reached, valve B is closed, A is opened, and C is adjusted for flow in direction 1 to 3. After all the bubbles in the line are flushed out, a syringe is filled at the point shown. This sample may be used as a dissolved O₂ standard with methylene blue or with other techniques such as gas chromatography.

The most convenient amino carboxylic acid for use with methylene blue is EDTA (ethylenediaminetetraacetic acid). A liter of methylene blue/EDTA solution is prepared from 800 ml of distilled water, 2.92 g of EDTA, 10M NaOH to adjust the pH to 10, 0.021 g methylene blue, and additional distilled water to make 1 liter of solution. Three milliliters of the reagent solution are added to each of three cuvettes filled with rubber septums. Each cuvette is degassed with argon for 10 to 15 minutes through a hypodermic needle. The degassed solution will be the colorless photo-reduced leuco form. It should be noted that since methylene blue is rapidly photo-reduced by light, care should be taken to avoid exposure to laboratory lighting.

Calibration solutions are prepared by injecting 10 to 250 μ l of the saturated standard solution into a series of reagent tubes. The absorbance at 660 nm

(which obeys Beer's law) is read and plotted on an absorbance versus concentration curve. Then the unknown sample is measured and the concentration obtained from the calibration curve.

Notes:

1. A technique for gas chromatographic analysis of dissolved oxygen has also been developed as part of this work.
2. Requests for further information may be directed to:
Technology Utilization Officer
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Reference: B73-10147

Patent status:

Inquiries concerning rights for the commercial use of this invention should be addressed to:

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